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Message:
Attached is prep procedure used for vegetable samples with exceptions noted in the Tech Memo. Method 6020 was used for analysis.

If you need a confirmation or any pages resent, please call our office.

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS

1. SCOPE AND APPLICATION

This procedure describes techniques used for sample preparation and acid digestion of biological tissue samples. This procedure is applicable to the analysis of biological tissue for heavy metals. The procedure provides a convenient and efficient digestion/dissolution technique which allows for the simultaneous or sequential analysis of the sample for metals. The digestates may be analyzed by graphite furnace atomic absorption (GFAA), Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). The procedure includes, but is not restricted to, the metals listed in Table 1.

2. METHOD SUMMARY

A representative tissue sample is Lyophilized, blended, then sub-sampled for microwave or conventional oven digestion. Oxidation is brought about by the use of concentrated nitric acid in a Teflon closed vessel. The digestate is then analyzed for metallic constituents by GFAA, ICP-OES, or ICP-MS methods.

3. INTERFERENCES

Refer to the determinative method for a discussion of interferences.

4. SAFETY

Nitric Acid is extremely corrosive. Care should be taken while working with this chemical. Personal protective equipment will include safety glass (with side shields), gloves and a lab coat. Follow normal precautions as per the CAS Safety Manual.

5. SAMPLE, COLLECTION, PRESERVATION AND STORAGE

Samples are typically collected in plastic containers. Sample may be refrigerated or frozen, depending on project requirements.

6. APPARATUS AND EQUIPMENT

6.1. Lyophilizing apparatus such as a LABCONCO Model 4.5 benchtop freeze dryer.

6.2. Microwave digestion apparatus including Teflon closed vessels fitted with rupture discs or equivalent pressure relief.

6.3. Conventional laboratory oven capable of precise temperature control at 105°C.

6.4. Analytical balance capable of weighing to 0.1 mg.

6.5. 50 mL graduated poly tubes.

7. STANDARDS AND REAGENTS

7.1. Reagent water - ASTM Type II water

7.2. Concentrated nitric acid.

7.3. 1,000 ppm stock metal standards for preparation of spike solution(s) to include analyte(s) of interest.

8. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

9. PREVENTATIVE MAINTENANCE

9.1. Routine cleaning of the sample handling and digestion apparatus is necessary. Refer to the SOP for Metals Laboratory Glassware Cleaning.

9.2. Record all maintenance and monitoring activities in a lab notebook.

10. PROCEDURE

10.1. Obtain a representative tissue sample that will yield ~ 200 mg of freeze-dried solids.

Note: This is designed to be a general guideline. Approximately 200 mg of dry sample is typically required to obtain the desired detection limits that often are necessary for tissue analysis.

10.2. Slice the sample into thin pieces prior to filling the drying vessel.

10.3. Cap the vessel and freeze the sample in a conventional freezer.

10.4. When the sample is frozen, remove from freezer and follow the manufacturer's instructions for operation of the freezer dryer.

10.5. Blend the dry solids to obtain a homogeneous sample. The sample may be stored dry until digestion. grind - mortar + pestle or blender

10.6. Microwave digestion option

10.6.1. Transfer 200 mg of dried sample, weighed to the nearest 0.1 mg, to a 50 mL decomposition vessel.

10.6.2. Add 3 mL concentrated nitric acid to the vessel.

Note: The procedure may be continued immediately with greater chance of rupturing the safety seal on the decomposition vessel than if the sample is allowed to stand for several hours, or more, pre-digesting at room temperature. Pre-digesting can also be enhanced by use of heat lamps.

10.6.3. Follow the manufacturer's instructions for the use of the decomposition vessels.

10.6.4. Place the vessel in the carrousel and expose at 1/2 power for 5 minutes.

10.6.5. Cool the vessel, open to relieve pressure and vent gases, then return to carrousel.

10.6.6. Note: perform this step in the hood.

10.6.7. 1 Expose at full power for 10 minutes.

10.6.8. Cool the vessel, open to relieve pressure and vent gases, then transfer to a volumetric container and dilute to 20 mL. The sample is ready for analysis.

√10.7. Conventional oven digestion option

10.7.1. Transfer 200 mg of dried sample, weighed to the nearest 0.1 mg, to a 50 mL decomposition vessel.

10.7.2. Add 3 mL concentrated nitric acid to the vessel.

Note: The procedure may be continued immediately with greater chance of rupturing the safety seal on the decomposition vessel than if the sample is allowed to stand for several hours, or more, pre-digesting at room temperature. Pre-digesting can also be enhanced by use of heat lamps.

10.7.3. Follow the manufacturer's instructions for the use of the decomposition vessels. Place the vessel in a conventional oven at 105°C for a minimum of 24 hours.

10.7.4. Cool the vessel, open to relieve pressure and vent gases, then transfer to a volumetric container and dilute to 20 mL. The sample is ready for analysis.

11. QA/QC REQUIREMENTS

11.1. If using the conventional oven option, monitor oven temperatures for each batch. Report all deficiencies to the Lab Manager; corrective action will be taken.

11.2. Prepare one blank per digestion batch, or per 20 samples, or per SDG , whichever is more frequent. Analyze at least one preparation blank per group of samples digested.

11.3. Prepare duplicates at 5% frequency or one per batch, whichever is more frequent.

11.4. Prepare one laboratory control sample at 5% frequency or one per batch, whichever is more frequent.

11.5. Prepare spikes at 5% frequency or one per batch, whichever is more frequent. Spikes should be added directly to the dry sample. Spike solutions should be multi-element with analyte concentrations high enough to minimize volume added to sample. The acid matrix of the spiking solution should be 20-50% nitric acid, if possible.

11.6. Analyze a standard reference material (SRM) at 5% frequency or one per batch, whichever is more frequent. SRM's should be representative of the tissue sample being analyzed.

12. REPORTING

12.1. Digestion data sheets, including weights and volumes used, are completed and a batch lot number is assigned and attached to the data sheet. The Manufacturer's lot number for the reagents used are added to the digestion data sheet. (See appendix A)

12.2. Spiking sheets are completed including all spike data and volumes of spiking solutions used. (See appendix A)

13. REFERENCES

Recommended Guidelines for Measuring Metals in Puget Sound Marine Water, Sediment, and Tissue Samples; April 1997.